Cylindrical Light Scattering Cell

L. P. WITNAUER AND H. J. SCHERR
Eastern Regional Research Laboratory and Department of Chemistry, Temple University,† Philadelphia, Pennsylvania*

ZIMM[1] has described a method for measuring high molecular weights by light scattering from solutions or suspensions. The method requires no assumption of a particle shape. It consists in measuring the intensity distribution of scattering over as great an angular range as possible and then extrapolating to zero angle. This paper describes the construction and performance of a cylindrical cell for making such measurements down to angles as low as 22° to the incident beam. The cell is designed specifically for the B-S photometer[2] but can be adapted for use with other instruments by making obvious modifications in the dimensions.

A scale drawing of the cell is shown in Fig. 1. The body of the cell is a 12-cm length of 41-mm o. d. standard wall Pyrex tubing. The tubing is free from obvious defects. The tube is ground lengthwise to give a pair of parallel windows, 8 mm wide, with both the inner and outer faces flat. One-half of the tube is frosted over its full inside length to reduce multiple reflections of stray light. A circular glass plate is fused to one end of the tube as a bottom. The bottom of the cell is cemented into a square plastic base which fits the stage of the B-S light-scattering photometer and aligns the entrance and exit faces with respect to the incident light.

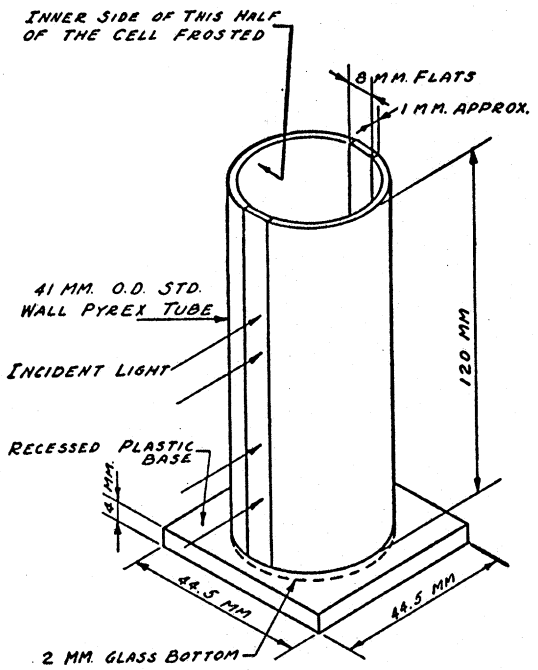

FIG. 1. Diagram of cylindrical light-scattering cell.

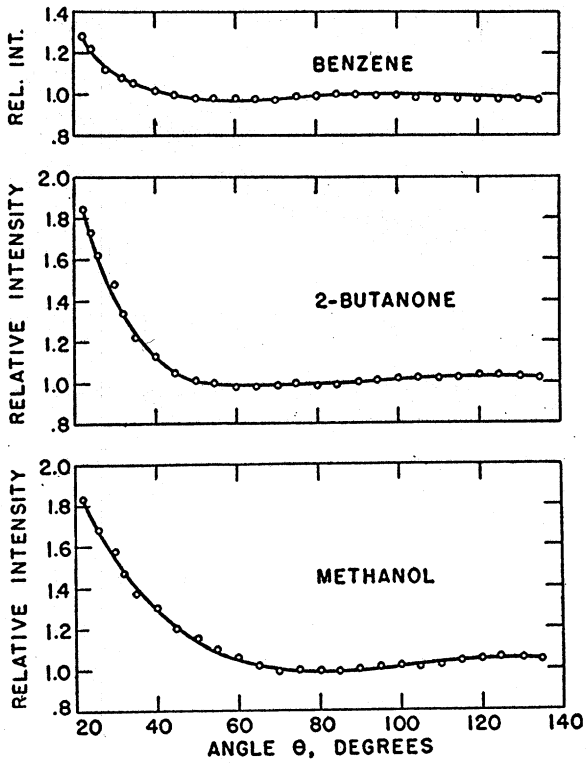

FIG. 2. Angular variation of scattered intensity from the cell containing benzene, 2-butanone, and methanol. θ is the angle of observation.

To produce the inner faces of the windows the glass tubing is rubbed on a brass lap with an abrasive mixed with glycerine or water. Grinding is done in three stages using three laps, each with a different abrasive. The abrasives were, successively, American Optical Company Numbers 180, 302, and 304 (cerium oxide). The laps were made from brass stock $\frac{3}{8}$ in.×1 in.×14 in., with the grinding surface reduced in width from $\frac{3}{8}$ in. to 8 mm. A series of grooves $\frac{1}{16}$ in. wide and $\frac{3}{8}$ in. apart is cut in the lap surface at an angle of 45° to the sides. One end of the lap is clamped in a vise, the tube slipped over the other end, and the grinding done with a back-and-forth stroke. The first or rough grinding operation is stopped when the curved inner surface of the tube is essentially flat. The second and third grinding operations using finer abrasives serve to smooth and flatten the inner faces and greatly facilitate the final polishing operation. The outer faces of the windows are

ground to the desired width on a rotating lap mounted on a vertical spindle. All window faces are polished with felt polishing cloth, the inner faces being done on a felt surfaced lap. The final thickness of the windows is approximately 1 mm.

The frosting of one-half of the tube is accomplished on a semi-cylindrical lap grooved similarly to the flat laps. The only abrasive employed is No. 180.

The B–S photometer is altered for use with the cell by replacing the normal 12-mm wide limiting apertures with 4-mm apertures, thus permitting measurements to be made down to 22° before the phototube views any illuminated portion of the incident face.

The cell was checked for distortion of the scattering envelope by measuring the light intensity over the range 135° to 22° for a solution of the sodium salt of fluorescein of concentration 1×10^{-4} mg/ml. This solution radiates with an intensity of the same order of magnitude as usually encountered in light-scattering determinations of molecular weights. A constant intensity (±1 percent) was obtained over the angular range after subtraction of the solvent blank and correcting for variation in the volume viewed by multiplication by the sine of the angle of observation.

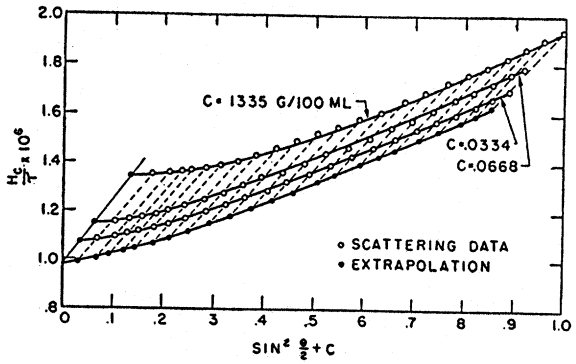

FIG. 3. Reciprocal turbidity plot for polystyrene in 2-butanone.

The most important requirement of a cell of this type is that the blank correction must not be excessive at low angles. Figure 2 shows the relative intensity of scattering corrected for volume viewed and normalized to unity at 90° for benzene, 2-butanone, and methanol. The values have also been corrected for changes in the state of polarization of the scattered light as a function of angle by multiplication by $1/(1+\cos^2\theta)$, where θ is the angle of observation, taking due account of the fact that the light scattered at 90° is incompletely polarized. The solvents were distilled into the scattering cell just before use.

The plots of Fig. 2 show that the blank correction is substantially constant down to an angle of 40° in the most favorable case and 60° in the least favorable one. Moreover, even at the smallest angle the increase in the blank correction is relatively small compared with the total scattering from solutions used in molecular weight determinations. This increase may be due in part to the observed presence of scintillating particles which could not be removed or to extraneous reflections.

A polystyrene fraction distributed through the International Union of Chemistry and described by Doty and Steiner[3] was selected to illustrate the use of the cell. The polystyrene was dissolved in 2-butanone and the solutions filtered through a Corning ultrafine sintered glass filter. The scattered intensity for 436 mμ plotted as described by Zimm[1] is given in Fig. 3. The value obtained for the molecular weight is 1.02×10^6, uncorrected for depolarization. This agrees well with the value 1.03×10^6 reported by Doty and Steiner,[3] and is further evidence for the satisfactory performance of the cell.

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† This work will be included as part of a dissertation to be submitted by Lee P. Witnauer to the Graduate School of Temple University in partial fulfillment of the requirements for the Ph.D. degree.

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[2] Brice, Halwer, and Speiser, J. Opt. Soc. Am. 40, 768 (1950).
[3] P. Doty and R. F. Steiner, J. Chem. Phys. 18, 1211 (1950).